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Short Communication

Study on controlling thermal expansion coefficient of ZrO_2–TiO_2 ceramic die for superplastic blow-forming high accuracy Ti–6Al–4V component

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ABSTRACT

The high temperatures and extended times involved with superplastic forming titanium mean that conventional die-steels are not suitable. Although dies manufactured from nickel-based alloys are used frequently, these are expensive, and more importantly, the coefficient of thermal expansion (CTE) of this materials are bigger than that of Ti–6Al–4V, which causes the dimensional inaccuracy between the dies and workpieces. The aim of this paper is to control the CTE of ZrO_2–TiO_2 ceramic die by adjusting the volume fraction of TiO_2 and the relative density, and then controlling its' CTE to be similar to Ti–6Al–4V alloy. In this paper, firstly, the relation between the expansion coefficient and the volume fraction of TiO_2 and ceramic relative density was investigated. The results showed that with the increment of the volume fraction of TiO_2, the expansion coefficient decreased. And with the rising of the relative density, the expansion coefficient increased. Secondly, based on the Turner Equation, a modified theoretical model was proposed in which the relative density of ceramic was considered. Using the model, the comparison was made between the experimental data and calculated data. The result showed that the linear CTE of ZrO_2–TiO_2 can be controlled by adjusting the volume fraction of TiO_2 and corresponding relative density. Thirdly, to evaluate the practicability of the ZrO_2–TiO_2 ceramic die on superplastic forming, the ZrO_2–TiO_2 ceramic die (volume fraction of TiO_2 is 27%, relative density is 93%) with the expansion coefficient ($8.92 \times 10^{-6}\,°C^{-1}$) similar to Ti–6Al–4V was fabricated by sintering at 1520 °C for 2 h. The experiment of superplastically forming Ti–6Al–4V using ZrO_2–TiO_2 cylinder ceramic die was carried out, also. The Ti–6Al–4V cylinder shows better shape retention, surface quality and high dimensional accuracy, and the ceramic die seems adequate for superplastically forming the high accurate Ti–6Al–4V.

1. Introduction

Superplastic forming (SPF) permits the production of complex shapes in sheet metal. Consequently a single SPF manufacturing process can produce complex structural components without the need for welding or riveting parts together [1]. Among superplastic materials, titanium alloys such as Ti–6Al–4V have been put into wide applications in the aerospace industry because of their superior properties such as a high strength-to-weight ratio, high temperature tolerance and excellent corrosion resistance [2–7]. Ti–6Al–4V used to be superplastically formed at temperature about 750–950 °C. This high forming temperature means that the conventional steel dies are not suitable. Up to date heat resistant cast steels, especially nickel–chromium cast steels, such as ZG35Cr24Ni7Si, are the normal solution for Ti-SPF-forming die manufacturing. However, dies manufactured from refractory steel are expensive and would not be capable of operating at temperatures in excess of about 1000 °C. Compared to steel dies, ceramic dies own better elevated temperature property, high hardness and stand wear and tear, they offer greater potential for extended die life and therefore lower power consumption during operation. More importantly, the thermal expansion coefficient (CTE) of refractory steel are far bigger than that of Ti–6Al–4V. This difference of CTE will cause the dimensional inaccuracy, especially to the bigger dimensional workpieces, because the linear contraction of dies is bigger than that of the Ti–6Al–4V workpieces when cooling after superplastic forming. To some workpieces with complex shapes, the plastic deformation can even arise when cooling. And also because of this difference, the Ti–6Al–4V workpieces are so difficult to remove from the female die, that the warping, even fracture often generates. It is obvious that the difference of CTE between the workpiece and die leads to considerable problems in engineering practice of superplastic forming. The combination of high temperature and suitable linear expansion coefficient points to the consideration of the ceramic die for superplastic forming. Refractory castables or ceramic dies have been investigated in order to offer alternatives to the SPF producers [8–12]. To the ceramic dies, if their CTE was matched to that of the Ti–6Al–4V, workpiece size will change to a little extent as it cools from the forming temperature (930 °C).

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In this paper, the influence of relative density and different volume fraction of TiO_2 on the linear CTE of ZrO_2–TiO_2 ceramic was investigated by dilatometer thermal expansion measurement. According to the results, a modified theoretical model based on Turner model was proposed in which the relative density of ceramic was considered. And also the comparison between experimental data and model predictions was performed. Based on the theoretical model, the ZrO_2–TiO_2 ceramic die with the matching linear CTE with Ti–6Al–4V alloy was fabricated. Using the optimal ZrO_2–TiO_2 ceramic die, the Ti–6Al–4V alloy cylinder was superplastically formed and the accuracy was analyzed.

2. Measuring CTE of ZrO_2–TiO_2 ceramic

2.1. Sintering ZrO_2–TiO_2 ceramic cylinder specimens

The trial ceramic die was prepared by sintering the green of the ZrO_2–TiO_2 with the Polyvinyl Alcohol (PVA) in the atmosphere. The addition of TiO_2 into ZrO_2 cannot only adjust the linear expansion coefficient but also prevent the chemical interreaction between the ceramic die and the Ti–6Al–4V alloy. Thus, commercial TiO_2 (chempurity 99%), supplied by the Tianjin No. 3 Chemical Reagent Factory, was added to ZrO_2. And also the PVA (m(PVA):m (H_2O = 7:100)) was added to the mixing powder as the ceramic binder in order to avoid the ceramic die cracking when sintered and enhance the moldability. The basic properties of ZrO_2 and TiO_2 are shown in Table 1.

To measure the linear expansion coefficient of ZrO_2–TiO_2 ceramic, the ceramic cylinder specimens of the size $\phi 10 \times 50$ mm were fabricated. The process consists of four steps. Firstly, the powders of ZrO_2 and TiO_2 were mixed in a vertical planetary ball milling using sintered carbide balls at room temperature. Then PVA was mixed into the milled mixture in agitator for 1 h at 120 °C. Secondly, the green of ZrO_2 and TiO_2 was pressed into the mould of cylinder (Fig. 1) at 100 °C. The semi-finished ceramic cylinder was taken out after cooling for 3 h at room temperature. Thirdly, debinding and pre-sintering were performed in heating-furnace from 25 °C to 1000 °C. Finally, the ceramic cylinder was sintered, the finished ZrO_2–TiO_2 cylinder is shown in Fig. 2b.

Table 1
Basic properties of ZrO_2 and TiO_2.

	Density (g/cm^3)	Young's modulus (GPa)	CTE[a] ($\times 10^{-6}$ °C^{-1})
3y–ZrO_2	5.75	205	9.6
TiO_2	4.2	282	8.2

[a] Note: at 930 °C.

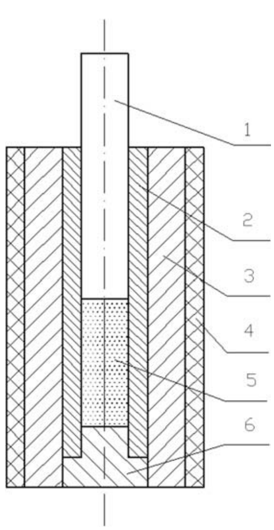

Fig. 1. Schematic diagram of the structure of the die. (1) Formed punch; (2) female die; (3) outer sleeve; (4) heating apparatus; (5) powder lot; (6) filling piece.

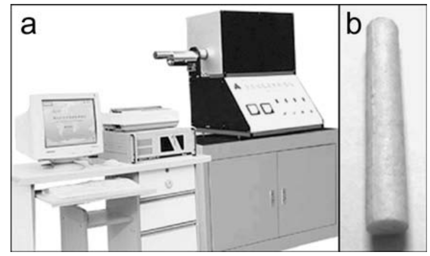

Fig. 2. The RPZ-03P thermal dilatometer (a) and ZrO_2–TiO_2 ceramic cylinder specimen (b).

2.2. Measurement of CTE of ZrO_2–TiO_2 ceramic specimen

The measurement of linear expansion coefficient of ZrO_2–TiO_2 ceramic has been performed with RPZ-03P thermal dilatometer (Fig. 2a). Because the optimal superplastic forming temperature of Ti–6Al–4V is 930 °C, the linear expansion coefficient was measured at temperature ranging from 200 °C to 930 °C. The heating rate was 5 °C/min and the measurement was conducted in air. The CTE measurement was parallel to the pressing direction.

2.3. Results and discussion

To determine the relation between the linear CTE and the content of TiO_2, five groups ZrO_2–TiO_2 cylinder specimens was sintered with different volume fraction of TiO_2 (0%, 15%, 30%, 40%, 55%, respectively). Fig. 3 shows the five curves of linear CTE of ZrO_2–TiO_2 ceramic at the temperature of 200–930 °C, the result shows that the linear CTE of ZrO_2–TiO_2 ceramic decreases with the increase of TiO_2 content. This phenomenon can be explained by the Turner Equation [13].

Because the Linear CTE of TiO_2 is smaller than ZrO_2, it is reasonable that the experimental Linear CTE of ZrO_2–TiO_2 decreases correspondingly with the increment of TiO_2.

The influence of the relative density on the linear expansion coefficient was also investigated. Four groups ZrO_2–TiO_2 cylinder specimens with 30% volume fraction TiO_2 were sintered at different temperatures (1550 °C, 1450 °C, 1400 °C, 1350 °C, respectively) and the relative density were 0.92, 0.81, 0.75 and 0.70 correspondingly. The results (Fig. 4) show that the linear CTEs of 200–930 °C

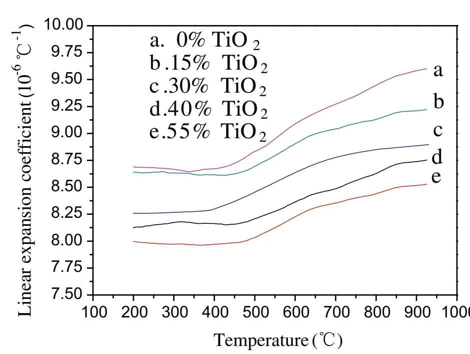

Fig. 3. The linear CTE of ZrO_2–TiO_2 ceramic with different volume fraction of TiO_2.

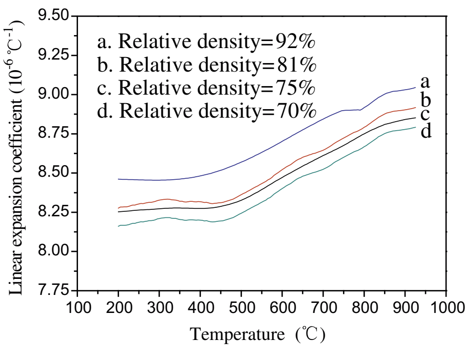

Fig. 4. The linear CTE of ZrO_2–TiO_2 ceramic with different relative density.

of ZrO_2–TiO_2 ceramic increase with the relative density rising. This is because the less the relative density is, the more the voids exist in the ceramic. When the temperature rises, the amplitude of material particle increases, but the expansion of material particle was partly absorbed by the voids in the ZrO_2–TiO_2 ceramic, leading to the overall expansion decreasing.

3. Modified theoretical model

3.1. Theoretical model equation

According to the results of the measurement of CTE, it is obvious that the linear CTE of ZrO_2–TiO_2 was not only affected by the volume fraction of TiO_2, but also by the relative density of ceramic. Because during the superplastic forming, the material was formed under a low pressure at the temperature close to 0.5 of the absolute melting temperature, it is not necessary for the ceramic die to own a very high strength. That means the ceramic die needs no absolute density. Turner Equation is valid for the sample without voids and free of thermal stresses, the deviation were often found between the experimental results and calculation [14,15]. So it is reasonable to consider the relative density into the model to predict the actual CTE of ZrO_2–TiO_2. Based on the Turner equation, a modified theoretical model was proposed as follows:

$$a = \sqrt[3]{d}\left(\frac{a_P V_P E_P + a_m V_m E_m}{V_P E_P + V_m E_m}\right) \tag{1}$$

where a is the linear expansion coefficient, d the relative density of the ceramic, V the volume fraction, E the elastic modular and P and m represent second-phrase ceramic and base ceramic, respectively.

3.2. Comparison between experimental data and model predictions

To fabricate the ZrO_2–TiO_2 ceramic die with the same expansion coefficient with Ti–6Al–4V ($8.92 \times 10^{-6}\,°C^{-1}$ at 930 °C), based on the theoretical model equation, the different volume fraction of TiO_2 and corresponding relative density can be calculated. According to the calculated results, the five groups ZrO_2–TiO_2 ceramic cylinders was pressed with different volume fraction of TiO_2 and sintered to corresponding relative density. Then the linear CTE of actual ceramic cylinder was measured. The average experimental data at 930 °C was compared to the modified model predictions (Fig. 5). The result shows that the experimental linear CTEs were approximately matched to the calculated data. And with the increase of the content of TiO_2, the relative density requested increases. It is obvious that the linear CTE of ZrO_2–TiO_2 can be controlled by adjusting the volume fraction of TiO_2 and corresponding relative density.

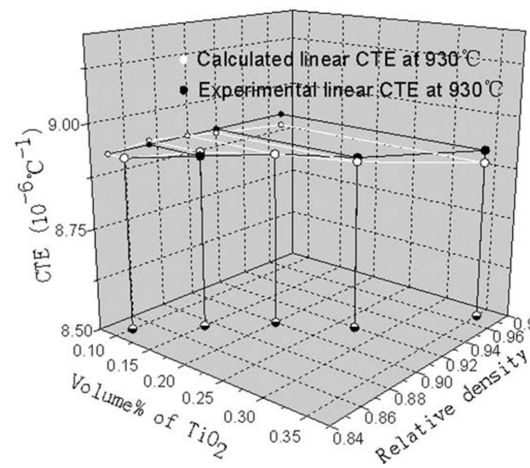

Fig. 5. Different volume fraction of TiO_2 and corresponding relative density for fabricating the optimal ZrO_2–TiO_2 ceramic die with same linear CTE with Ti–6Al–4V (8.92×10^{-6} °C at 930 °C).

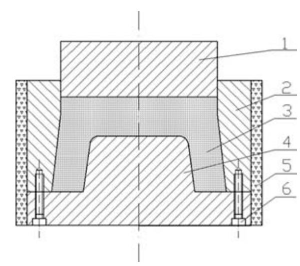

Fig. 6. The steel model using to form the semi-finished ZrO_2–TiO_2 deep cylinder ceramic die. (1) Pressure head; (2) external mold; (3) mixing powder; (4) internal mold; (5) heating apparatus; (6) screw bolt.

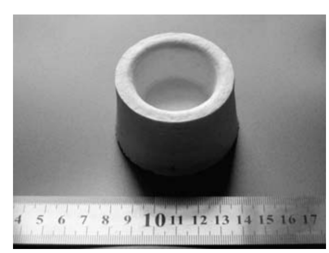

Fig. 7. The finished ZrO_2–TiO_2 deep cylinder ceramic die.

4. Superplastic forming Ti–6Al–4V deep cylinder by ZrO_2–TiO_2 ceramic die with the same linear CTE with Ti–6Al–4V

4.1. The ZrO_2–TiO_2 deep cylinder ceramic die

The ceramic die used in superplastic forming requires high hot strength to resist cracking and fracture, low hot deformation and minimal chemical interaction with the metal to be formed. According to this, the sample, whose volume fraction of TiO_2 is 27% and corresponding relative density is 93%, was chosen to fabricate the ceramic die for superplastic forming. In this process, the deep cylinder ZrO_2–TiO_2 ceramic die was formed by the steel die (Fig. 6). Firstly, a double layers of cling film were covered on the steel model in order to separate the ceramic die easily. Secondly, the mixing powder was compressed into the cavity bit by bit at 120 °C till all the powder was used up. Then allow them to set at room temperature. Thirdly, 30 min later, the mixing powder turned to be

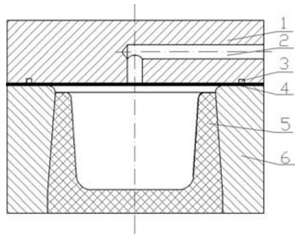

Fig. 8. Schematic diagram of superplastic forming Ti–6Al–4V deep cylinder with ceramic die. (1) Blank holder; (2) air inlet hole; (3) blank slot; (4) Ti–6Al–4V sheet; (5) ceramic die; (6) female die.

Fig. 9. The Ti–6Al–4V deep cylinder superplastically formed by ZrO_2–TiO_2 ceramic die.

hardened and owns the cylinder shape. After separating it from the Steel die and removing the cling film, debinding and pre-sintering were performed in heating-furnace from 25 °C to 1000 °C for 48 h. Next, the ceramic die was sintered at 1520 °C for 2 h in the hot sintering furnace to enhance the strength and get the relative density of 92%. Fig. 7 shows the finished ZrO_2–TiO_2 deep cylinder ceramic die.

4.2. Superplastic forming Ti–6Al–4V using ZrO_2–TiO_2 cylinder ceramic die

In order to evaluate the practicability of the ZrO_2–TiO_2 ceramic die on superplastic forming, the experiment of superplastically forming Ti–6Al–4V using cylinder ceramic die was carried out. The schematic diagram of superplastic blow forming was showed in Fig. 8. This test was performed on the 1000KN superplastic forming machine. The SPF machine consists of a heater stove which operates at 300 V using a 3-Phase power supply, a 1000KN oil hydraulic press and a water cooling system. The temperature in heater stove was measured by the electric therm-couple. After fixing the ceramic die to the female die and putting the specimen on the female die, the blank holder was pressed onto the Ti–6Al–4V sheet in the heater stove of SPF machine and the steel air pipe welded on the air inlet hole was screwed to the gas-pressure meter of the nitrogen cylinder. The Ti–6Al–4V sheet was heated at a rate of 15 °C min^{-1} up to 930 °C and held for 30 min before blow forming. Then the nitrogen gas was controlled by using gas-pressure meter to blow the Ti–6Al–4V plate onto the ceramic die surface. The gas pressure applied on the plate was increased gradually up to 1.5 MPa. In order to achieve complete adaptation of the Ti–6Al–4V sheet to the ceramic die surface, a dwell time of 20 min was used before it was allowed to cool. After cooling in the atmosphere, the Ti–6Al–4V deep cylinder (Fig. 9) was easily separated from the ZrO_2–TiO_2 ceramic die surface because of their identical expansion coefficient. The Ti–6Al–4V cylinder shows better shape retention, surface quality and high dimensional accuracy, confirming that the ceramic owns enough mechanical strength and no chemical reaction with the Ti–6Al–4V at the temperature of 930 °C. Consequently the ceramic die seems adequate for superplastically forming Ti–6Al–4V with high accuracy, and the trials have confirmed the potential of the ZrO_2–TiO_2 ceramic die.

5. Conclusions

1. The linear CTE of ZrO_2–TiO_2 ceramic shows dependence not only on the content of TiO_2 but also on the relative density of ceramic. The CTE decreased with the increase of the volume fraction of TiO_2, and increased with the increase of the relative density.
2. The modified theoretical model $\left(a = \sqrt[3]{d}\left(\frac{a_p V_p E_p + a_m V_m E_m}{V_p E_p + V_m E_m}\right)\right)$ was proposed, in which the relative density was considered. Using the model, the calculated linear CTE of ZrO_2–TiO_2 ceramic were matched to the experimental data.
3. The ZrO_2–TiO_2 deep cylinder ceramic die (volume fraction of TiO_2 is 27% and relative density is 93%), with linear expansion coefficient similar to that of Ti–6Al–4V, was fabricated. The Ti–6Al–4V sheet was superplastically formed by means of such a die. The Ti–6Al–4V cylinder shows better shape retention, surface quality and high dimensional accuracy.

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